An Accelerated Thermal Oxidative Aging Method for Asphalt Binder under Atmospheric Oxygen-Rich Conditions

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Abstract

An exploratory method based on gaseous oxidation was developed for asphalt binder to accelerate thermo-oxidative aging under conditions representative of the field environment. An oxygen-rich atmosphere containing OX and NOX (X=1–3) was generated using a self-developed gas aging vessel (GAV), while atmospheric pressure and realistic summer pavement temperatures were maintained. Rheological and chemical changes were characterized using dynamic shear rheometry and Fourier transform infrared spectroscopy, respectively. The rheological assessment included the G-R parameter, complex modulus, creep rate, and stiffness modulus. Additionally, and aging-related functional group indices were determined for asphalt binder. After GAV aging, an increase in complex modulus was accompanied by improved resistance to permanent deformation. In contrast, fatigue resistance decreased at intermediate-temperatures, while susceptibility to cracking increased at low-temperatures. Compared with standard laboratory short-term aging, GAV aging produced substantially greater changes in both chemical and rheological properties. After 72 h of GAV aging, the sulfoxide index, aging index, stiffness modulus at -18 °C, and G-R parameter increased by 70%, 49%, 36%, and 24%, respectively. The proposed GAV method can accelerate the thermo-oxidative aging of asphalt binder while maintaining temperature and pressure conditions close to those encountered in the field.

Keywords

Asphalt Binder; Thermal-Oxidative Aging; Oxygen-Rich Atmosphere; Rheological Properties; Chemical Properties.

1. Introduction

Asphalt binder is extensively employed in in pavement engineering because of its favorable engineering properties [1-3]. However, it is continuously exposed to heat, oxygen, ultraviolet (UV) radiation, and moisture during mixture production and pavement service. These environmental factors induce volatilization, oxidation, and polymerization, resulting in alterations to the binder's chemical composition and molecular structure. Consequently, the binder's physical characteristics and rheological properties deteriorate, potentially contributing to pavement distress [4-7]. Binder aging significantly influences pavement performance.

Laboratory testing is routinely employed to evaluate the susceptibility of asphalt binders to aging. Compared with natural aging, accelerated laboratory aging can reproduce substantial property changes within a much shorter period. Standard laboratory procedures employ the rolling thin-film oven test (RTFOT) for short-term aging and the pressure aging vessel (PAV) test for long-term aging [8,9]. Glover et al. [10] reported that aging thin binder films at 60 °C under atmospheric pressure for approximately 35 days produced rheological and carbonyl changes comparable to those obtained after 20 h of PAV aging. Similar results were reported by Zelalem et al. [11] based on changes in the carbonyl area. Nevertheless, the high temperature and pressure applied during PAV aging may induce reactions that do not occur under actual pavement conditions. There has been debate about whether laboratory aging tests can accurately reproduce the oxidative aging of asphalt binder under field conditions [12,13]. As noted by Anderson, the PAV was developed primarily to produce substantial oxidation within a short period rather than to reproduce field aging conditions directly [14].

Field aging is dominated by relatively slow oxidation. In addition to molecular oxygen, highly reactive atmospheric species, including ozone and nitrogen oxides, may contribute to oxidation of the pavement surface [15]. As an organic material, asphalt binder is vulnerable to attack by reactive oxygen and nitrogen species such as O_X and NO_X (X=1-3) [16]. Nitrogen oxides emitted from vehicle exhaust can also participate in photochemical reactions that promote ozone formation [17]. In order to accelerate oxidation under field relevant temperature and pressure conditions, Steiner et al. [18] developed an accelerated aging device. Strongly oxidizing gases were used while maintaining realistic temperature and pressure conditions. Exposure of asphalt mixtures for 4 days at 60 °C produced effects comparable to those of combined RTFOT and PAV treatment, whereas 6 days of exposure resulted in substantially greater hardening. However, this procedure was designed to age asphalt mixtures rather than asphalt binders [19].

This study proposes an approach to accelerate asphalt binder aging while maintaining field-relevant temperature and pressure conditions. It focuses on accelerating the overall oxidation process by modifying the gaseous environment. A gas aging vessel (GAV) operating at atmospheric pressure was designed. Asphalt binder was aged for different durations under atmospheric pressure in an oxygen-rich environment. Its rheological and chemical changes were characterized using a dynamic shear rheometer (DSR). Chemical changes were examined by Fourier transform infrared (FTIR) spectroscopy. To reduce test duration, RTFOT was com-pared as the standard to show the aging degree of GAV. The proposed method is expected to provide a more realistic approach for simulating thermo-oxidative aging and to enhance assessments of long-term aging in asphalt binders.

2. Materials and Methods

2.1 Materials

This study employed an unmodified asphalt binder. The properties of the used asphalt binder are shown in Table 1. Samples were subjected to RTFOT aging in accordance with ASTM D2872. As show in Fig. 1, the GAV aged samples were prepared. To reduce the duration of aging and obtain sufficient samples for testing, the film thickness of the samples was 0.8mm.

Table 1. The properties of asphalt binder

Properties	Results	Standards
Penetration/0.1mm	69	ASTM D5
Softening point/°C	47.5	ASTM D36
Ductility/cm	43	ASTM D113
Dynamic viscosity/Pa·s	203	ASTM D4402

2.2 GAV Aging Method

As shown in Fig. 1, compressed air was passed through an ozone generator to produce an oxygen-rich atmosphere containing ozone and nitrogen oxides. The generated gas was then introduced into a vacuum drying oven for asphalt binder aging. The aging temperature was maintained at 60 °C under atmospheric pressure. Asphalt binders were aged for 24 and 72 h, respectively. This oxygen-rich environment was employed to accelerate was employed to under field-relevant temperature and pressure conditions.

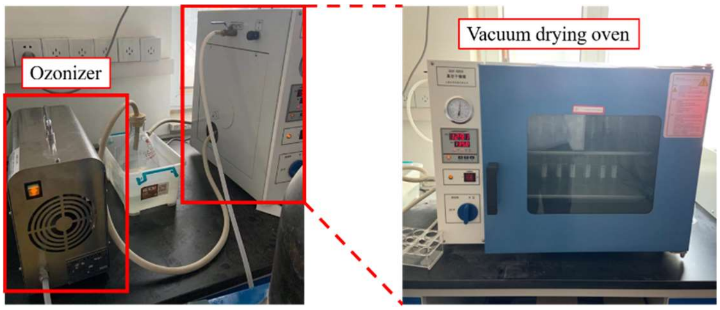

Fig. 1 Gas aging vessel

2.3 Dynamic Shear Rheological Test

The DSR was used to assess binder rheology. For each test, two replicate specimens were prepared. Testing was repeated if the relative deviation between the results was greater than 10%.

The frequency sweep test was conducted using parallel plates with a diameter of 25 mm and a plate separation of 1 mm. The angular frequency was varied between 0.1 and 100 rad/s. Tests were conducted under strain control at 35, 45, and 55 °C in strain-controlled mode with the strain amplitude fixed at 1%. The complex modulus curves for different test temperatures were horizontally shifted according to the time–temperature superposition principle. These curves were then combined to form a master curve at the reference temperature. The master curve was employed to describe binder rheology across a broad range of frequencies. The Williams-Landel-Ferry (WLF) nonlinear equation [20] was used to determine the temperature shift factors.

The frequency sweep test was conducted using 8 mm parallel plates. Tests at 5, 15, and 25 °C were conducted with a strain amplitude of 1%. A master curve of complex modulus was constructed from the resulting data. The complex modulus and phase angle at 15 °C and 0.005 rad/s were obtained to calculate the G-R parameter [21].

The frequency sweep measurements were performed using a 4 mm aluminum parallel plate geometry with a plate separation of 2 mm. Tests were conducted at -6, -12, and -18 °C by sweeping the angular frequency from 0.2 to100 rad/s with a strain amplitude of 1%. Based on the method proposed by Sui et al. [22], the stiffness modulus (S) and creep rate (m) at the three test temperatures were calculated from the 4 mm DSR test results. These parameters were employed to assess the binder's deformation response and stress relaxation behavior at low temperatures.

2.4 Fourier Transform Infrared Spectroscopy Test

FTIR analysis was conducted with a Spectrum II infrared spectrometer. A spectral resolution of 0.5 cm^{-1} was used, and 32 scans were acquired across a wavenumber interval of 4000-500 cm^{-1}. Binder spectra were collected using an attenuated total reflectance (ATR) accessory was used to obtain the spectra of the asphalt binders. The test procedure proposed by Mirwald et al. [23] was adopted. Each binder was tested five times for statistical analysis. A background scan was performed before each measurement.

3. Results and Discussion

3.1 Evaluation of Aging Effects based on Rheological Parameters

Asphalt binder exhibits typical viscoelastic characteristics, and its complex modulus is particularly responsive to aging-induced alterations in rheological behavior. Through the frequency scanning test at 35℃, 45℃ and 55℃, the master curves of complex modulus of asphalt binder were shown in Fig. 2.

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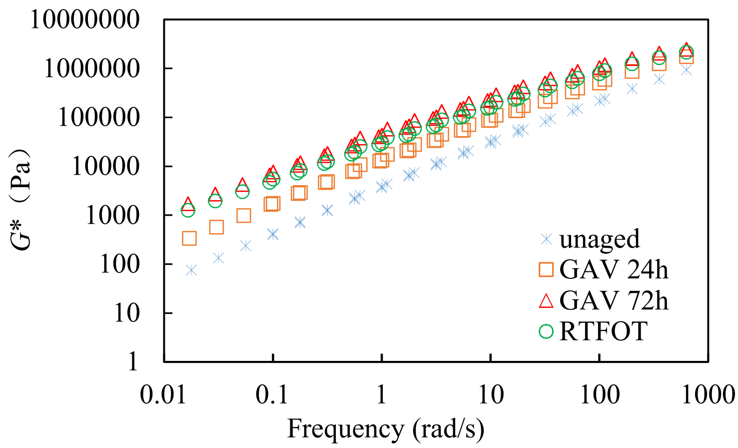

Fig. 2 The complex modulus master curve of asphalt binder before and after aging

As shown in Fig. 2, the complex modulus increased continuously with increasing frequency for all asphalt binders. Binder deformation was therefore restricted, resulting in a higher apparent stiffness. Accordingly, greater resistance to permanent deformation can be expected under high-frequency loading conditions. Aging caused an obvious upward shift in the master curves of complex modulus across the full frequency range. The lowest complex modulus was observed for the unaged binder, reflecting the highest deformability. After GAV aging, higher complex modulus values were observed with longer exposure durations. The modulus after 72 h of GAV aging was consistently higher than that after 24 h, confirming the progressive hardening of asphalt binder during prolonged aging. The 72 h GAV-aged asphalt binder also exhibited a slightly higher complex modulus than the RTFOT-aged asphalt binder over most of the frequency range. This suggests that 72 h of GAV aging produced a slightly stronger hardening effect than RTFOT aging.

To verify the proposed aging method, the effects of oxidation and temperature were separated and evaluated. The same unaged asphalt binder was placed in the GAV device, and nitrogen was used to replace the oxidizing gas to prevent oxidation. The aging temperature and duration were kept identical to those used in the previous tests. Frequency sweep tests were performed prior to and following the aging treatment.

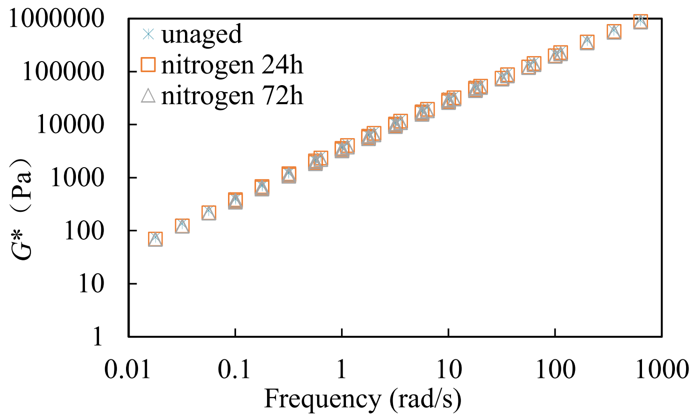

Fig. 3 The complex modulus master curve of asphalt binder (nitrogen)

The results are shown in Fig. 3. The complex modulus after 24 and 72 h of aging under a nitrogen atmosphere was very close to the value measured for the unaged binder. The observed differences remained small as the aging duration increased. These findings suggest thatthermal exposure alone had little influence on binder rheology under the selected conditions. Therefore, the marked rise in complex modulus observed in Fig. 2 was mainly attributed to oxidation induced by the mixed gas in the GAV system rather than to temperature alone.

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To quantitatively assess the progression of binder aging, an aging index (I_a) was formulated using the complex modulus. The extent of binder aging at different exposure times was assessed using this index. The aging index was calculated using Eq. (1).

$$I_a = \frac{G_A* - G_U*}{G_U*} \tag{1}$$

Where G_A* = the complex modulus of asphalt binder after aging; G_U* =the complex modulus of unaged asphalt binder.

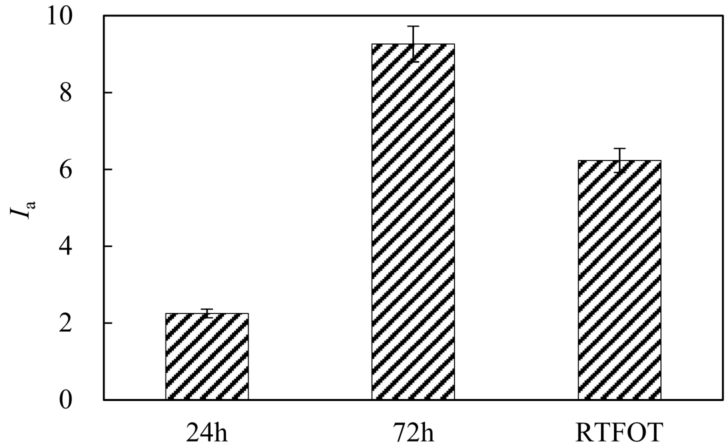

Fig. 4 Aging index based on complex modulus

As shown in Fig. 4, the aging index after 72 h of GAV aging was 4.12 times that after 24 h. It was also 49% higher than that obtained after RTFOT aging. These findings show that the extent of binder aging increased markedly as GAV exposure was prolonged.

The G-R parameter is widely employed to assess binder resistance to fatigue cracking. It is particularly responsive to aging-induced alterations in rheological behavior. Therefore, it can effectively reflect the deterioration of fatigue performance during aging. The G-R parameters of asphalt binder were shown in Fig. 5.

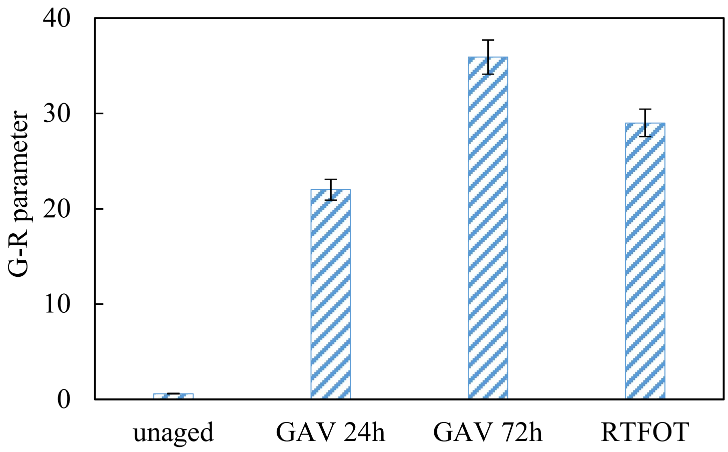

Fig. 5 The G-R parameters of asphalt binder before and after aging

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As shown in Fig. 5, the G-R parameter increased progressively with increasing GAV aging time. Higher G-R values are usually associated with increased binder brittleness and reduced resistance to fatigue cracking. The G-R parameter after 72 h of GAV aging exceeded the value obtained after RTFOT treatment by 24%. This finding suggests that prolonged exposure to the GAV environment caused more severe deterioration in fatigue-related rheological properties. With increasing aging time, the asphalt binder became stiffer and more brittle, and its capacity to dissipate stress during cyclic loading decreased. Consequently, the binder became more prone to fatigue cracking. These results further suggest that an oxygen-rich environment can accelerate oxidative aging and promote aging-induced embrittlement. Therefore, prolonged exposure to such conditions may make asphalt pavements more susceptible to fatigue cracking.

Binder resistance to cracking at low-temperatures is commonly assessed using stiffness modulus and creep rate. These parameters are highly sensitive to aging-induced changes in binder stiffness and stress relaxation behavior. The stiffness modulus and creep rate of the asphalt binder before and after aging at different temperatures are shown in Fig. 6.

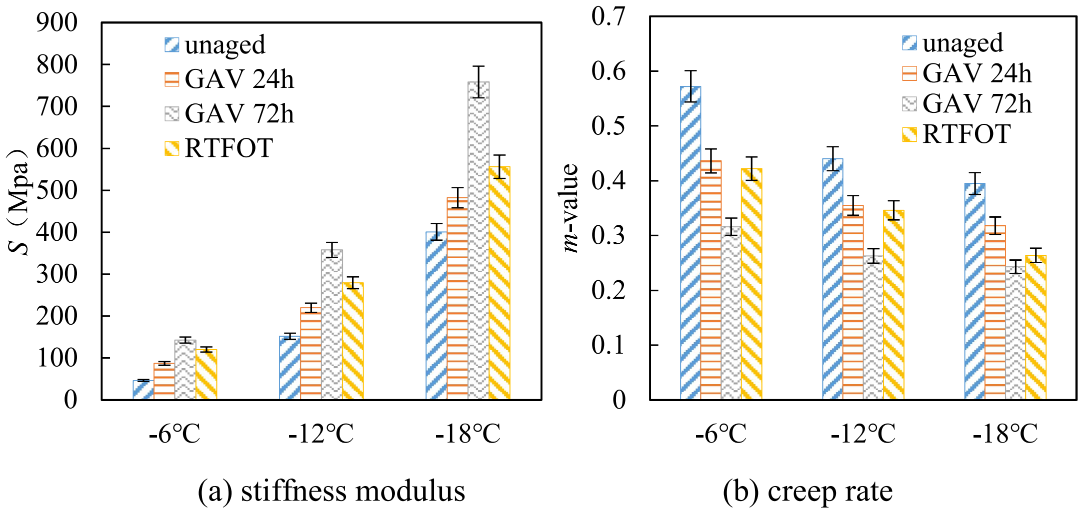

Fig. 6 The stiffness modulus and creep rate of asphalt binder before and after aging

As shown in Fig. 6, the *S* value increased, whereas the *m*-value decreased with increasing GAV aging time at all temperatures. These changes indicate that GAV aging increased binder stiffness while reducing the capacity for stress relaxation. Consequently, the asphalt binder became more brittle and its cracking resistance at low-temperatures gradually deteriorated. This effect became more pronounced after 72 h of GAV aging.

A clear temperature dependence was also observed. The stiffness modulus increased markedly as temperatures fell from -6 to -18 °C, while creep rate showed a continuous decline. This behavior was ascribed to reduced mobility of binder molecules under colder conditions. The elastic response gradually became dominant, resulting in higher stiffness and poorer stress relaxation. Therefore, asphalt binder became more prone to low-temperature cracking as the temperature decreased.

The values obtained after RTFOT aging were generally between those obtained after 24 and 72 h of GAV aging. At -6, -12, and -18 °C, the stiffness modulus after 72 h of GAV aging was 18%, 28%, and 36% higher than that after RTFOT aging, respectively. Meanwhile, the corresponding *m*-values after 72 h of GAV aging were lower than those after RTFOT aging. These results indicate that 72 h of GAV aging caused more severe deterioration in low-temperature rheological properties than RTFOT aging. The increasing difference at lower temperatures further suggests that GAV aging has a stronger effect on the binder’s susceptibility to cracking under colder conditions.

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3.2 Evaluation of Aging Effects based on Chemical Properties

Overall, aging led to binder stiffening and embrittlement, while its deformation capacity andstress relaxation ability were reduced. The binder also became more susceptible to fatigue cracking and low-temperature cracking. These rheological changes were mainly associated with oxidation-induced variations in binder chemistry. Oxidation represents one of the main reactions involved in this process and generates oxygen-containing functional groups. FTIR can effectively characterize these chemical changes and is therefore widely used to evaluate the extent of binder oxidation. In this study, FTIR analysis was employed to investigate the evolution of oxygen-containing functional groups during the aging process. The FTIR spectra of the asphalt binder before and after aging are shown in Fig. 7.

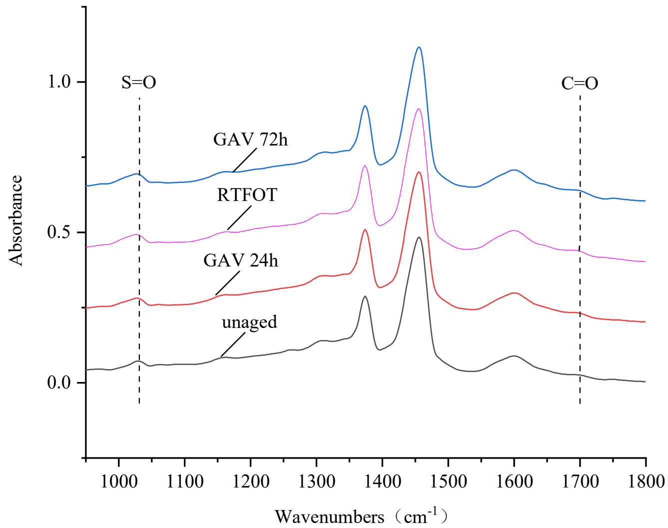

Fig. 7 The FTIR spectra of asphalt binder before and after aging

As shown in Fig. 7, the positions and shapes of the absorption peaks of the aged asphalt binder were similar to those of the unaged asphalt binder. This indicates that GAV aging did not produce any readily discernible new functional groups. Nevertheless, changes in the absorption intensity and peak area caused by aging could not be directly distinguished from the spectra. Therefore, quantitative analysis of characteristic oxygen-containing functional groups was required to further evaluate the chemical changes during aging.

The main oxidation products formed during the aging of asphalt binders, whether virgin or modified, include carbonyl (C=O) and sulfoxide (S=O) groups, with characteristic absorption bands at approximately 1700 cm^{-1} and 1030 cm^{-1}, respectively. The characteristic peaks of methyl and methylene groups, located between 1200 $cm^{-1}$and 1550 cm^{-1}, showed no significant changes during aging. Therefore, they are commonly used as reference peaks for quantifying aging-related functional groups. Variations in the content of these groups can be quantitatively evaluated using their characteristic peak areas. The carbonyl index ($I_{C=O}$) and sulfoxide index ($I_{S=O}$) were calculated using Eq. (2) and Eq. (3).

$$I_{C=O} = \frac{A_{C=O}}{A_{\text{ref}}} \tag{2}$$

$$I_{S=O} = \frac{A_{S=O}}{A_{\text{ref}}} \tag{3}$$

Where $A_{C=O}$ = the area of carbonyl characteristic peak; $A_{S=O}$ = the area of sulfoxide characteristic peak; A_{ref} = the area of saturated C-H bond bending vibration absorption peak.

The quantitative FTIR analysis of asphalt binder was based on the Beer-Lambert law. The baseline method and the analysis functions in OMNIC software were used to determine the characteristic peak areas. The carbonyl index and sulfoxide index of the asphalt binder before and after aging are presented in Table 2.

Table 2. Carbonyl index and sulfoxide index of different aged asphalt binder

	Unaged	GAV 24h	GAV 72h	RTFOT
$I_{C=O}$	0.0032	0.0041	0.0060	0.0057
$I_{S=O}$	0.0119	0.0197	0.0416	0.0244

As shown in Table 2, the indices for carbonyl and sulfoxide groups rose as GAV aging was prolonged, indicating progressive binder oxidation. Compared with the unaged asphalt binder, $I_{C=O}$ increased by approximately 28% after 24 h of GAV aging and by 88% after 72 h. The $I_{C=O}$ value after 72 h of GAV aging also exceeded that obtained after RTFOT treatment by about 5%. This finding suggests that carbon-containing components were gradually oxidized and converted into carbonyl-containing compounds during GAV aging. The relatively small increase after 24 h suggests that carbonyl formation was limited at the early stage of aging.

A more pronounced variation was observed for the sulfoxide index. Compared with the unaged asphalt binder, $I_{S=O}$ increased by approximately 66% after 24 h and 250% after 72 h of GAV aging. Moreover, the $I_{S=O}$ value after 72 h of GAV aging was approximately 70% higher than that after RTFOT aging. These results indicate that sulfur-containing components actively participated in the oxidation process and were progressively converted into sulfoxide-containing compounds. The much larger increase in $I_{S=O}$ than in $I_{C=O}$ also suggests that the sulfoxide group was more sensitive to the GAV aging process under the investigated conditions.

In summary, the formation and accumulation of oxygen-containing polar groups increased the polarity of asphalt binder and strengthened intermolecular interactions, thereby restricting molecular mobility and increasing binder stiffness and brittleness. This chemical evolution was consistent with the rheological results. With increasing GAV aging time, higher values were observed for the complex modulus, stiffness modulus, and G-R parameter, whereas creep rate declined. These changes indicated progressive deterioration in deformation capacity and stress relaxation ability, together with poorer fatigue resistance and greater susceptibility to low-temperature cracking. Meanwhile, consistent aging trends were observed in the indices describing binder chemistry and rheology, confirming a close association between chemical composition and rheological response. This agreement across the two sets of indicators demonstrates that the proposed oxygen-rich GAV method can effectively reproduce the progressive oxidative aging of asphalt binder. It provides a feasible approach for evaluating aging-induced changes in binder properties.

4. Summary

The objective of this study was to develop an accelerated thermo-oxidative aging method using an oxygen-enriched GAV environment. DSR and FTIR tests were used to characterize the rheological and chemical changes in asphalt binder, respectively. It also verified the feasibility of the proposed accelerated aging method. RTFOT aging was used as a reference for comparison. The main conclusions are as follows.

(1) The GAV method was demonstrated to be effective for accelerating the thermo-oxidative aging of asphalt binder. After 72 h of GAV aging, the aging index was 49% higher than that obtained after

RTFOT aging. Similarly, the sulfoxide index increased by 70% compared with RTFOT-aged asphalt binder.

(2) With increasing GAV aging time, the complex modulus of asphalt binder increased, indicating enhanced stiffness and deformation resistance. However, aging increased binder brittleness and reduced its fatigue resistance and low-temperature cracking resistance.

(3) No new functional groups were formed during GAV aging. Both the carbonyl and sulfoxide indices increased markedly with aging time. Compared with the unaged asphalt binder, the sulfoxide index increased by 132% and 250% after 24 and 72 h of GAV aging, respectively.

(4) This study provides a preliminary investigation of accelerated thermo-oxidative aging of asphalt binder using the GAV method. Future work will focus on optimizing the GAV parameters and incorporating additional aging factors, such as UV radiation. These improvements are expected to provide a more realistic and time-efficient simulation of the long-term aging of asphalt binder.

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